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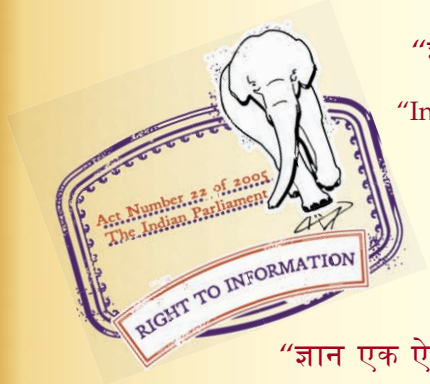

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Indian Standard

METHODS OF TEST FOR PULP

PART XVIII DETERMINATION OF CHLORINE CONSUMPTION (DEGREE OF DELIGNIFICATION)

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Indian Standard

METHODS OF TEST FOR PULP

PART XVIII DETERMINATION OF CHLORINE CONSUMPTION (DEGREE OF DELIGNIFICATION)

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Indian Standard

METHODS OF TEST FOR PULP

PART XVIII DETERMINATION OF CHLORINE CONSUMPTION (DEGREE OF DELIGNIFICATION)

0. FOREWORD

0.1 This Indian Standard (Part XVIII) was adopted by the Indian Standards Institution on 8 October 1979, after the draft finalized by the Paper and Its Products (Excluding Packaging Materials) Sectional Committee had been approved by the Chemical Division Council.

0.2 The method given in this standard for measuring the degree of delignification of pulp by measuring its chlorine consumption under specified conditions has the advantage over the method for determination of kappa number given in IS : 6213 (Part X)-1975* that it is not restricted to pulps obtained in yields under 75 percent. It has been found experimentally that there is a linear relationship between the chlorine consumption and the total lignin content of pulp. This relationship is independent of the method used in the manufacture of pulp.

0.3 In the formulation of this standard, considerable assistance has been derived from IS0 3260-1975 'Pulps — Determination of chlorine consumption (degree of delignification)', issued by the International Organization for Standardization.

0.4 In reporting the results of a test or analysis, made in accordance with this standard, if the final value, observed or calculated, is to be rounded off, it shall be done in accordance with IS : 2-1960†.

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1. SCOPE

1.1 This standard (Part XVIII) prescribes the method for determination of the degree of delignification of **pulp** by measuring its chlorine consumption. This method is applicable to all kinds of pulp.

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*Methods of test for pulp: Part X Detrrmination of kappa number,
†Rules for rounding off numerical values *(revised)*.

2. TERMINOLOGY

2.0 For the purpose of this standard the following definition shall apply.

2.1 Chlorine Consumption of Pulp — The amount of active chlorine consumed by the pulp under the conditions specified in this standard. The chlorine consumption is expressed as a percentage by mass.

3. QUALITY OF REAGENTS

3.1 Unless otherwise specified pure chemicals and distilled water *(see* IS : 1070-1977*), freshly boiled and cooled, shall be employed in the tests.

NOTE — 'Pure chemicals' shall mean chemicals that do not contain impurities which affect the results of analysis.

4. PRINCIPLE

4.1 A test portion of pulp is treated for 15 minutes, at a temperature of 27 ± 1°C, with chlorine generated by acidification of a sodium hypochlorite solution. The residual chlorine, which shall be more than 50 percent of the amount added, is determined by iodometric titration. Chlorine consumption so obtained is corrected to the consumption at constant concentration of available chlorine.

5. REAGENTS

5.1 Sodium Hypochlorite Solution — Containing about 20 g of active chlorine per litre and of a total alkalinity corresponding to a *p*H of 12.0 ± 0.5 measured with a glass electrode.

5.2 Hydrochloric Acid — 4 N, obtained by adding 100 ml of hydrochloric acid of relative density 1.19 g/ml, to 200 ml of water.

5.3 Potassium Iodide Solution— 1 N, containing 166 g of potassium iodide per litre.

5.4 Standard Sodium Thiosulphate Solution — 0.2 N. The normality shall be known to ± 0.000 4 N.

5.5 Starch Indicator Solution— 2 g/1.

6. APPARATUS

6.1 High Speed Wet Disintegration Apparatus — The apparatus shall be such that it disintegrates the pulp completely with a minimum of damage to the fibres, for example, a kitchen mixer or a similar apparatus.

*Specification for water for general laboratory use (*second revision*).

6.2 Apparatus for Determination of Chlorine Consumption- It shall be as shown in Fig. 1, consisting of a thick-walled conical flask and a separating funnel.

6.2.1 *Thick-Walled Conical Flask* — It shall be of 750 ml capacity with a standard ground joint.

6.2.2 *Separating Funnel* — It shall be of 50 to 100 ml volume, with standard ground joints and a glass socket.

6.3 Motor Driven **Coated Magnetic St-irrer** — It shall provide efficient stirring when the magnet and the motor table are approximately 40 mm apart.

6.4 Water-Bath — Capable of maintaining a temperature of 27 ± 1°C for at least 20 minutes and provided with a support for the flask.

6.5 Vacuum Pump

6.6 Stop-Watch

7. PREPARATION OF SAMPLE

7.1 Air-Dried Pulp Sheets — Tear 3 to 10 g of the pulp into small pieces.

7.2 Screened **Slush Pulps** — Make a 3 to 10 g air-dry pad by filtering on a Buchner funnel, avoiding any loss of fibres. Air-dry the pad and tear it into small pieces.

7.3 Unscreened Pulps — If the pulp sample is taken from unscreened pulp which is normally screened before bleaching or other processing, then the shives and knots shall be removed from the sample by screening. The method of screening shall be stated in the test report and shall be chosen to give results similar to those obtained by the industrial screening of the pulp. Complete the preparation of the screened pulps as in 7.2.

8. PROCEDURE

8.1 Preparation of Test Portion — Before weighing the test portions, condition the samples for not less than 20 minutes in the atmosphere near the balance. Weigh out 500 ± 5 mg of the pulp. At the same time, weigh out a separate portion for the determination of dry matter content.

8.2 Determination

8.2.1 Disintegrate the test portion in the disintegrator in 250 ml of water at 26 to 27°C until free from fibre clots and large bundles. Transfer the disintegrated test portion to the reaction flask using 135 ml of water

to rinse the disintegrator. Place the flask on the support in the water-bath and start the stirrer. Connect the separating funnel and evacuate the flask by means of the vacuum pump. Close the stop-cock of the funnel, remove the socket and add 10 ml of the hydrochloric acid to the funnel.

8.2.2 Suck down the acid without admitting air and start the stop-watch simultaneously. Rinse the funnel with 10 ml of water and suck it down. Pipette 15.0 ml of the sodium hypochlorite solution into the funnel and suck it down after exactly 2 minutes. Do not stop the watch at this stage. Rinse the funnel with 5 ml of water and suck it down. Add 20 ml of the potassium iodide solution to the funnel and suck it down exactly 17 minutes after adding the hydrochloric acid. Rinse the funnel with 50 ml of water, suck it down and shake the flask to dissolve gaseous chlorine. Add 50 ml of water to the funnel and suck it down; leave the stop-cock open and remove the funnel. Titrate with the sodium thiosulphate solution using starch as the indicator. Record the consumption as V_1 ml.

8.2.3 Perform a blank determination using the same procedure and record the consumption as V_2 ml.

NOTE — For pulps with a very low chlorine consumption, use a smaller volume of sodium hypochlorite solution and increase the volume of water in proportion. Carry out the blank determination with the same volumes of sodium hypochlorite and water. For titration, use a standard volumetric sodium thiosulphate solution of lower normality than that stated in **5.4.**

8.2.4 Carry out two determinations.

9. CALCULATIONS

9.1 Calculate the fraction c of added chlorine not consumed in the determination as follows:

$$c = \frac{V_1}{V_2}$$

where

c = fraction of added chlorine not consumed in the determination;

V_1 = the volume, in ml, of standard volumetric thiosulphate solution consumed in the titration of the test portion; and

V_2 = the volume, in ml, of standard volumetric thiosulphate solution consumed in the titration in the blank determination.

If c is found to be less than 0.5, repeat the determination with a smaller test portion. If c is greater than 0.5, obtain the correction factory f from Table 1.

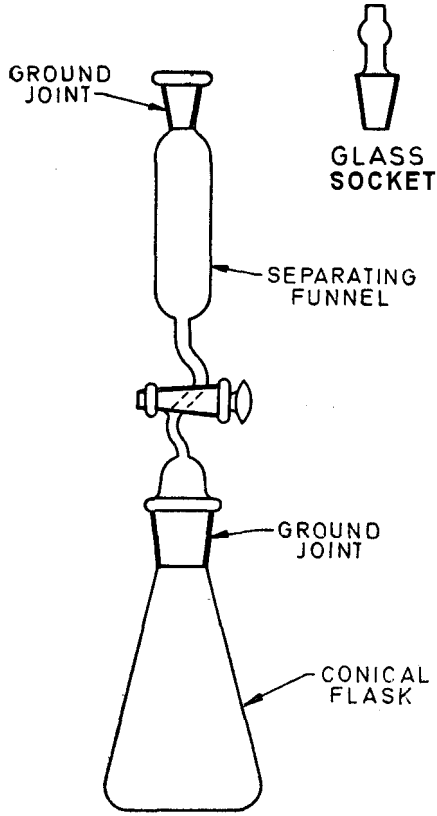

FIG. 1 APPARATUS FOR DETERMINATION OF THE CHLORINE CONSUMPTION OF PULP

TABLE 1 FACTOR *f* FOR CORRECTION OF CHLORINE CONSUMPTION

(Clause 9.1)

c		0.01	0.02	0.03	0.04	0.05		0.07	0.08	0.09
0.5	1.193	1.187	1.181	1.175	1.170	1.164	1.159	1.154	1.148	1.143
0.6	1.139	1.134	1.129	1.124	1.120	1.115	1.111	1.107	1.103	1.098
0.7	1.094	1.061	1.087	1.083	1.079	1.075	1.072		1.065	1.061
0.8	1.058	1.055	1.051	1.048	1.045	1.042	1.039		?? 1.033	1.030
0.9	1.027	1.024	1.021	1.018	1.016	1.013	1.010		??? 1.005	1.003

9.2 Calculate the chlorine consumption *X,* expressed as a percentage by mass, as follows:

$$X = \frac{3{\cdot}546 f (V_2 - V_1) T}{m}$$

where

X = chlorine consumption, percent by mass:

V_2 = the volume, in ml, of standard volumetric thiosulphate solution consumed in the titration in the blank determination;

V_1 = the volume, in ml, of standard volumetric thiosulphate solution consumed in the titration of the test portion;

T = the normality of the standard volumetric sodium thiosulphate solution; and

m = the mass, in g, of the test portion, calculated on an oven-dry basis.

NOTE — For calculating the chlorine consumption *X,* the expression

$$f = \tfrac{1}{2} \left(1 + \frac{V_2}{V_2 - V_1} \ln \frac{V_2}{V_1} \right)$$

has been used. It has been derived on the basis of certain assumptions that are generally accepted in the theory of pulp chlorination. It has been proved experimentally and is valid only if *c* is greater than 0.5.

Report the result as the mean of the two determinations to three significant figures.

10. TEST REPORT

10.1 The test report shall include the following information:

a) All the indications necessary for complete identification of the sample;

b) The results and the form in which they are expressed;

c) In the case of unscreened pulp, the method of screening;

d) Any unusual features observed in the course of the test; and

e) Any operations not specified in this standard or regarded as optional, which may have affected the results.

INDIAN STANDARDS
ON

METHODS OF TEST FOR PAPER AND ALLIED PRODUCTS

IS:

1060 (Part I)-1966 Methods of sampling and test for paper and allied products, Part I *(revised)*

1060 (Part II)-1960 Methods of sampling and test for paper and allied products, Part II

1060 (Part III)-1969 Methods of sampling and test for paper and allied products, Part III

2188-1962 Methods of test for ??? for electrical purpose

4006 (Part I)-1966 Methods of test for paper and pulp based packaging materials, Part I

4006 (Part II)-1972 Methods of test for paper and pulp ??? packaging materials, PartII

4006 (Part III)-1978 Methods of test for paper and pulp ??? packaging materials, Part III

5285-1969 Methods ??? test for fibre analysis paper and board

6213 Methods of test for pulp
(Part I)-1971 Water solubility of pulp
(Part II)-1971 Determination of freeness of pulp
(Part III)-1971 Determination of alpha, beta, and gamma cellulose in pulp
(Part IV) -1971 Determination of viscosity ??? pulp
(Part V)-1971 Solubility ??? pulpin ??? percent caustic ??? solution
(Part VI)-1971 Copper number of pulp
(Part VII)-1971 Ash content in pulp
(Part VIII)-1973 Beating, sheet making, preparation of hand sheets and testing
(Part IX)-1973 Bleach requirement ??? preparation of hard sheets for optical tests of pulp
(Part X)-1975 Determination of the kappa number
(Part XI)-1975 Determination of acid insoluble ash
(Part XII)-1975 Determination of calcium content
(Part XIII)-1975 Determination ??? copper
(Part XIV)-1975 Determination of iron
(Part XV) -1975 Determination of manganese
(Part XVI)-1978 Dirt count
(Part XVII)-1978 Determination ??? saleble mass ??? pulp

INTERNATIONAL SYSTEM OF UNITS (SI UNITS)

Base Units

QUANTITY	UNIT	SYMBOL
Length	metre	m
Mass	kilogram	kg
Time	second	s
Electric current	ampere	A
Thermodynamic temperature	kelvin	K
Luminous intensity	candela	cd
Amount of substance	mole	mol

Supplementary Units

QUANTITY	UNIT	SYMBOL
Plane angle	radian	rad
Solid angle	steradian	sr

Derived Units

QUANTITY	UNIT	SYMBOL	DEFINITION
Force	newton	N	1 N = 1 kg.m/s^2
Energy	joule	J	1 J = 1 N.m
Power	watt	W	1 W = 1 J/s
Flux	weber	Wb	1 Wb = 1 V.s
Flux density	tesla	T	1 T = 1 Wb/m^2
Frequency	hertz	Hz	1 Hz = 1 c/s (s^{-1})
Electric conductance	siemens	S	1 S = 1 A/V
Electromotive force	volt	V	1 V = 1 W/A
Pressure, stress	pascal	Pa	1 Pa = 1 N/m^2

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